Comparing the influence of ultrasonic and microwave pre-treatment on the solubilisation and semi-continuous digestion of waste activated sludge.

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Abstract
Anaerobic digestion is a well-known technique for the recovery of energy from waste activated sludge. To increase the biodegradability of the sludge and to enhance the digestion efficiency, pre-treatment methods are of great importance. In this study, ultrasound and microwave pre-treatment were compared, through the use of three pilot scale semi-continuous digesters with a solids retention time of 20 days. During the experiment, one reactor was fed with untreated sludge (blank), one with ultrasound pre-treated sludge and one with microwave pre-treated sludge (both treated with a specific energy of 2100 kJ/kg sludge). (Organic) dry matter, COD, carbohydrates and proteins were analysed during digestion. The solubilisation of the organic material was the most effective by the ultrasound pre-treatment. The biogas production, which is the result of a change in sludge composition, is the highest in the microwave pre-treated digester. This results in an increased biogas production of 27% (i.e. 0.105 L/g ODS, blank 0.085 L/g ODS), compared to a 23% (i.e. 0.108 L/g ODS) increase by the ultrasound pre-treated digester. From the energy balance it is clear that both pre-treatments are economically not feasible.
Keywords
Anaerobic digestion, waste activated sludge, pre-treatment, ultrasound, microwave, semi-pilot scale

INTRODUCTION

Anaerobic digestion is widely applied as a sludge treatment step with one of its main advantages the production of an energy-rich biogas to generate heat and electricity. One of the major drawbacks of anaerobic digestion, however, is the limited conversion of the organic matter and the long retention time (15 to 20 days), as a result of the presence of rigid and persistent organic structures (Appels et al., 2010; Demirel and Scherer, 2008).

It is generally agreed upon that the first step of the anaerobic digestion, the hydrolyis, is rate-limiting (Thiem et al., 2001, Ghyoot and Vertraete, 1997). Here, complex macromolecular structures (such as extracellular polymeric substances) are converted into simple, soluble organics. By applying pre-treatment techniques, this step can be facilitated, which results in higher degradation efficiencies and shorter retention times.

The ultrasonic (US) treatment (20-40 kHz) is based on the cavitation effect resulting in hydrodynamic shear (Khanal et al., 2007). Microwave (MW) irradiation as a pre-treatment technique mainly causes thermal hydrolysis by heating the sludge, but also athermal effects have been described (Eskicioglu et al., 2007).

The main aim of this paper is to compare these two pre-treatment methods with each other, for organics solubilisation and biogas production during semi-continuous digestion of waste activated sludge.

MATERIALS AND METHODS

Ultrasound (US) and microwave (MW) pre-treatment

For each treatment, 500 g of sludge were subjected to ultrasound waves (100W for 8 min) or introduced in the MW oven (MW irradiated at 800W for 1 min). The ultrasonic waves were applied using a US horn with a maximum power output of 150W (Bandelin Sonopuls HD 3200); for

microwave application, sludge samples were placed in a microwave oven (Sharp R-212; 2.45 GHz) with a maximum power output of 800W. The following equation was used to calculate the total amount of energy (SE) supplied to the sludge: $SE = \frac{P \cdot t}{V}$ (with P = power (W); t = treatment duration (s); V = treated sludge volume (L)). A total energy of 2100 kJ/kg sludge was applied in both treatments.

Semi-continuous anaerobic digestion

Three pilot scale semi-continuous digesters (50 L working volume each) were run in parallel at mesophilic conditions (37°C) for 67 days. Twice a week, secondary sludge samples were taken from the thickening table of the WWTP of Mechelen-Noord (Belgium), pre-treated and used to feed the digesters. One digester was fed with untreated sludge (blank) as a reference, while the others were fed with US and MW pre-treated sludge, respectively. Buffer tanks (16 L) were available on the pilot scale installation to store the sludge before insertion in the digesters. Every 8 hours, 833 mL of (un)treated sludge was (automatically) fed to the digesters, leading to a hydraulic retention time (HRT) of 20 days.

Measurements and analysis

The dry solids (DS) and organic dry solids (ODS) content were determined as described in the Standard Methods (APHA, 2006). pH was measured by a pH electrode (Mettler Toledo). The COD was determined using Nanocolor® COD 1500 test tubes (Macherey-Nagel) in a digital photometer Nanocolor® 500D (Macherey-Nagel). The measurement of the carbohydrates was based on the Anthrone method (Gerhardt et al., 1994) and the amount of proteins was analysed by using the Bicinchoninic Acid Method (Smith et al., 1985). All analyses were carried out on both the sludge and the supernatant to identify total and soluble fraction of each parameter.

RESULTS AND DISCUSSION

Sludge solubilisation by US and MW pre-treatment

Properties of blank and pre-treated sludge are listed in Table 1, all averaged over the 16 sludge samples taken during the 67 days test. No significant difference was observed for the DS and ODS concentration before and after treatment, hence water evaporation, mineralisation and volatilisation of (organic) material during the US and MW treatment were limited. Also the total concentration of COD, carbohydrates and proteins remains approximately unchanged.

Table 1. Average values of several composition variables for untreated, ultrasound and microwave pre-treated sludge

	Blank		US pre-treated		MW pre-treated	
	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation
DS (g/kg)	43.6	7.3	42.1	6.9	41.3	7.1
ODS (g/kg)	30.8	5.2	29.0	5.1	29.4	5.3
COD (mg O_2/L)	40981	10184	42306	6158	43931	10450
sCOD (mg O_2/L)	104	24	1923	954	226	168
Carbs (mg Glu-eq/L)	4670	325	4860	448	5049	458
sCarbs (mg Glu-eq/L)	41	2.09	480	17.6	55	4.91
Prot (mg BSA-eq/L)	21267	1490	21073	1138	20772	1319
sProt (mg BSA-eq/L)	162	12	1317	130	254	9

Both US and MW treatment did significantly change the soluble organics concentration. This is reflected in an increase in the concentration of the soluble COD (sCOD), soluble carbohydrates (sCarbs) and the soluble proteins (sProt).

The untreated sludge has a very low sCOD/COD ratio. This may be related to the freshness of the sludge. The samples were taken directly after the thickening table so there was no time for natural solubilisation of the sludge.

The US treatment resulted in an average increase of sCOD, sCarbs and sProt by 1749%, 1071% and 713%, respectively. The MW treatment resulted in an average increase of sCOD, sCarbs and sProt by 117%, 34% and 57%, respectively. Relatively spoken, more carbohydrates than proteins are solubilised during US treatment and more proteins than carbohydrates are solubilised during MW treatment. It is appealing that although the same energy was applied to the sludge by both pre-treatments, significant differences in solubilisation were observed.

Anaerobic digestion

The total DS removal yield, after 67 days, decreased from 37% to 34% due to US treatment and increased from 37% to 45% due to MW treatment.

For the US pre-treatment, there was an increase in removal of COD by 2% and a decrease in ODS, carbohydrates and proteins removal by 6%, 4% and 2%, respectively. The MW pre-treatment causes an increase in removal of ODM, COD, carbohydrates and proteins by 4%, 15%, 7% and 11%, respectively.

After the US pre-treatment, a higher concentration of organic material is released into the soluble phase (Table 1) compared to the MW treatment. The removal yield after US pre-treatment of (O)DS, COD, carbohydrates and proteins is lower then the removal yield of the MW pre-treatment. This results in a higher amount of biogas produced by MW pre-treated reactor (Fig 1).

The pH of the three reactors remains approximately constant over time. This observation proves that all the reactors are working stable and no acidification takes place.

Biogas production

The biogas production for the digesters is depicted in Figure 1. It was observed that the biogas production in the US (0.105 L/g ODS) and MW (0.108 L/g ODS) pre-treated digester was higher compared to the blank (0.085 L/g ODS). Over the entire period of 67 days, an average increase of 23% was observed for the US pre-treated digester and 27% for the MW pre-treated digester. The methane concentration in the biogas from all three the reactors showed not many differences and remained constant at approximately 64%.

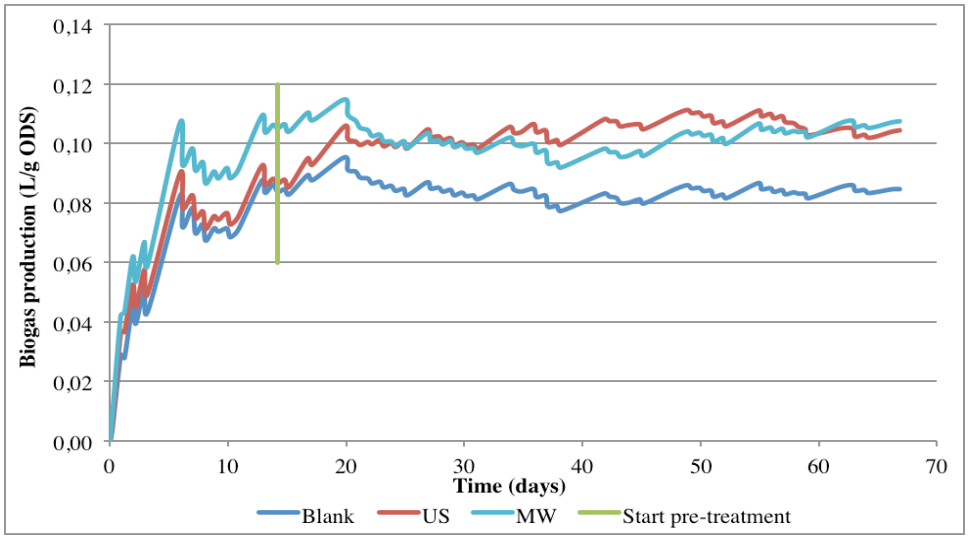

Figure 1. Biogas production (L/g ODS) of the blank, the US and MW treated digester

The US pre-treatment releases more organic material into the soluble phase than the MW pre-treatment. However, the biogas production is somewhat higher in the MW pre-treated digester. This suggests that the sole use of sCOD as an indicator for the improvement of digestion efficiency is not conclusive. Also other parameters need to be taken into account.

Economic evaluation

Table 2 shows the energy content of the extra biogas produced from US and MW-treated sludge and the energy needed for the pre-treatment.

Table 2. Energy balance (per g ODS fed to the reactor)

	US	MW
Average increase in biogas production (L)	0.020	0.023
Energy content of extra biogas (kJ)	0.459	0.530
US and MW energy applied (kJ)	3.109	3.109
Net energy production (kJ)	-2.650	-2.579

Both for the US pre-treatment as for the MW pre-treatment, there is no positive energy gain. The increased biogas production due to the US or MW pre-treatment does not provide enough energy to maintain the US and MW installation. The critical biogas enhancement value to achieve a positive energy gain would be 0.218 L/g ODS.

CONCLUSION

Pre-treatment can enhance the anaerobic digestion efficiency of waste activated sludge. Different types of pre-treatment methods are already proposed in the literature. In this paper the application of an US and MW pre-treatment is compared. The release of organic material into the soluble phase was larger for an ultrasound than for a microwave pre-treatment. The degradation of organic material in the digesters was higher for MW pre-treated digester resulting in a larger biogas production (27%) compared to the US pre-treated digester (23%). From the energy balance it is clear that both pre-treatments are economically not feasible.

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REFERENCES

APHA, 2006 Standard methods for the examination of water and wastewater. 18th ed.: American Public Health Association, Washington DC.

Appels L., Degrève J., Van der Bruggen B., Van Impe J. and Dewil R. 2010 Influence of low temperature thermal pre-treatment on sludge solubilisation, heavy metal release and anaerobic digestion. *Bioresource Technology*, **101**, 5743-5748.

Demirel B. and Scherer P. 2008 The role of acetotrophic and hydrogenotrophic methanogens during anaerobic conversion of biomass to methane: a review. *Reviews in Environmental Science and Biotechnology*, **7**, 173-190.

Eskicioglu, C., Terzian, N., Kennedy, K.J., Droste, R.L., Hamoda, M. 2007 Athermal microwave effects for enhancing digestibility of waste activated sludge. *Water Research*, **41**, 2457-2466.

Gerhardt, P., Murray, R.G.E., Wood, W.A., Krieg, N.R. 1994 Methods for general and molecular bacteriology, ASM, Washington DC.

Ghyoot W. and Verstraete W. 1997 Anaerobic digestion of primary sludge from chemical pre-precipitation. *Water Science and Technoogyl*, **36**(6-7), 357–65.

Khanal, S.K., Grewell, D., Sung, S., van Leeuwen (Hans), J. 2007 Ultrasound application in wastewater sludge pretreatment: A review. *Critical reviews in environmental science and technology*, **37**(4), 277-313

Smith, P.K., Krohn, R.I., Hermason, G.T., Mallia, A.K. 1985 Measurement of protein using Bicinchoninic acid. *Analytical Biochemistry*, **150**, 76-85.

Tiehm A., Nickel K., Zellhorn M.and Neis U. 2001 Ultrasonic waste activated sludge disintegration for improving anaerobic stabilization. *Water Research,* **35**(8), 2003–2009.